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GB 0318942.0

By virtue of a direction given under Section 30 of the Patents Act 1977, the application is proceeding in the name of:

ACCENTUS PLC,
329 Harwell,
DIDCOT,
Oxfordshire,
OX11 0QJ,
United Kingdom

Incorporated in the United Kingdom,

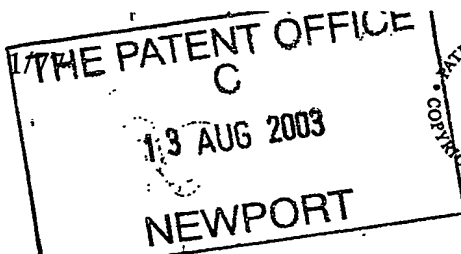
[ADP No. 08132243003]

and

FMC CORPORATION,
1735 Market Street,
Philadelphia,
Pennsylvania 19103,
United States of America

Incorporated in USA - Delaware,

[ADP No. 00462036009]



13AUG03 E829808-1 002834
P01/7700 0.00-0318942.0

Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

13 AUG 2003

The Patent Office

Cardiff Road
Newport
South Wales
NP10 8QQ

1. Your reference 15794 TpCm

2. Patent application number
(The Patent Office will fill in this part)

0318942.0

3. Full name, address and postcode of the or of each applicant (underline all surnames)

AEA Technology Batteries Systems Limited
329 Harwell
Didcot
Oxfordshire OX11 0QJ

FMC Corporation
1735 Market Street
Philadelphia
PA 19103
USA

Patents ADP number (if you know it)

FILED 22/1/04
SECTION 8559585001

00462036009

If the applicant is a corporate body, give the country/state of its incorporation

England and Wales

Delaware, USA

4. Title of the invention Process for producing an electrode

5. Name of your agent (if you have one)

Clare Josephine TALBOT-PONSONBY
Accentus plc
Patents Dept
329 Harwell
Didcot
Oxfordshire OX11 0QJ

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Patents ADP number (if you know it)

08132243001

6. If you are declaring priority from one or more earlier patent applications, give the country

Country

Priority application number
(if you know it)

Date of filing
(day / month / year)

7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application

Number of earlier application

Date of filing
(day / month / year)

8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

Yes

- a) any applicant named in part 3 is not an inventor, or
 - b) there is an inventor who is not named as an applicant, or
 - c) any named applicant is a corporate body.
- See note (d))

Patents Form 1/77

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Continuation sheets of this form

Description

11

Claim(s)

4

Abstract

1

Drawing(s)

1

10. If you are also filing any of the following, state how many against each item.

Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

1

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11.

Josephine T-P
CLARE JOSEPHINE TALBOT-POWSONBY
Agent

I/We request the grant of a patent on the basis of this application.

Signature

Date

12-8-03

MANSFIELD Peter Turquand

on behalf of AEA Techno
Battery Systems Ltd

12. Name and daytime telephone number of person to contact in the United Kingdom

Frances Esplin - 01235 43 2037

by a Power of Attorney
dated 15 May 2003.

Warning

After an application for a patent has been filed, the Comptroller of the Patent Office will consider whether publication or communication of the invention should be prohibited or restricted under Section 22 of the Patents Act 1977. You will be informed if it is necessary to prohibit or restrict your invention in this way. Furthermore, if you live in the United Kingdom, Section 23 of the Patents Act 1977 stops you from applying for a patent abroad without first getting written permission from the Patent Office unless an application has been filed at least 6 weeks beforehand in the United Kingdom for a patent for the same invention and either no direction prohibiting publication or communication has been given, or any such direction has been revoked.

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DUPLICATE

Process for producing an Electrode

The present invention relates to a process for
5 producing an electrode and electrodes produced by that
process, a process for producing a separator for a cell
and separators produced by that process and cells
comprising the electrodes and/or separators.

10 For many years it has been known to make cells with
lithium metal anodes, and cathodes of a material into
which lithium ions can be intercalated or inserted. Such
cells may use, as electrolyte, a solution of a lithium
salt in an organic liquid such as propylene carbonate,
15 and a separator such as filter paper or polypropylene.
More recently the use of a solid-state ion-conducting
polymer has also been suggested as an electrolyte. For
secondary or rechargeable lithium cells, the use of
lithium metal anodes is unsatisfactory as problems arise
20 from dendrite growth and electrolyte decomposition on
freshly deposited lithium. The elimination of this
problem is now possible by employing a material able to
intercalate lithium ions reversibly at very low voltages,
such as graphite, leading to so-called "lithium-ion", "
25 rocking-chair", or "swing" lithium rechargeable
batteries. These lithium cells operate on the principle
that they contain not lithium metal, but lithium ions
which are rocked back and forth between two intercalation
materials during the charging and discharging parts of
30 the cycle.

Conventional lithium ion cell technology uses a
carbon composite anode and a lithium metal oxide (usually
cobalt) composite cathode. During the formation cycle
35 (first charge), a solid electrolyte interface (SEI) layer
is produced on the surface of the carbon particles. This

layer greatly diminishes any further electrochemical reduction of the electrolyte, and allows the cell to operate for hundreds of cycles. However, producing the SEI layer consumes some of the lithium from the cathode. 5 This lithium is no longer available for reaction, and is termed the irreversible capacity of the anode.

US 2002/0119373 discloses the use of finely divided lithium powder in the anode of a cell. This has the 10 advantage of compensating for the irreversible capacity of the anode due to the formation of the solid electrolyte interface.

However, the stabilised lithium powder disclosed in 15 US 2002/0119373 reacts with solvents that are typically preferred for cell fabrication. In particular it is not compatible with N-methyl pyrrolidinone (NMP), dimethyl formamide (DMF), and dimethyl acetamide (DMA) which are the preferred solvents for the preferred binder 20 polyvinylidene fluoride (PVdF). Styrene butadiene rubbers and other similar binders may also be used in lithium ion cell anodes. These materials are commonly used as an aqueous suspension. When used as an aqueous suspension, these binders are also incompatible with 25 stabilised lithium powder, as the powder reacts with water.

It is therefore necessary to find an alternative way of incorporating lithium powder into a cell.

30

Accordingly, the present invention provides a process for producing an electrode which comprises forming an electrode precursor comprising a layer comprising an intercalation material, and then applying 35 particles to the surface of the electrode precursor wherein the particles comprise a material which

intercalates into the intercalation material of the electrode. The present invention also provides an electrode comprising an intercalation material and a surface coating of particles comprising a material which
5 intercalates into the intercalation material.

The electrode precursor is made of an intercalation material into which the material of the particles intercalates. The electrode that is formed may be a
10 cathode or an anode. Where the electrode is an anode, the intercalation material is a material which has a low voltage relative to lithium, for example one or more of carbonaceous materials, silicon, silicon containing materials such as silicon dispersed in carbon, tin, tin
15 oxides, composite tin alloys, and lithium metal nitrides. Where the electrode is a cathode one or more of lithium metal oxides and lithium metal phosphates such as LiCoO_2 , LiMn_2O_4 and LiFePO_4 .

20 The particles are preferably a finely divided powder, more preferably particles with a mean particle size of less than about 20 μm .

The particles comprise a material which intercalates
25 into the intercalation material. The particles are preferably lithium particles, more preferably stabilised lithium particles. In one embodiment of the present invention the particles are a mixture particles of a material which intercalates into the intercalation
30 material and another material, such as a mixture of lithium particles and carbon particles, and again the particles are preferably stabilised lithium particles. Methods for producing stabilised lithium particles are described in US 5,567,474, US 5,776,369 and US 5,976,403.

Where the particles are lithium particles, they may provide all of the lithium required by the cell. However as lithium has a low density it would tend to require a relatively thick surface layer to provide all of the lithium in this manner. Alternatively, the lithium particles may provide only some of the lithium provided by the cell, for example to compensate for the irreversible capacity of the anode.

10 The particles may be applied to the surface of any type of electrode precursor such as a composite electrode precursor formed from a mixture comprising an active material, binder, and solvent, an extruded electrode precursor comprising an active material and
15 binder/adhesive but no solvent, an electrode precursor formed by electrodeposition from a plating solution or an electrode precursor formed by surface coating techniques such as sputtering or chemical vapour deposition. Where the electrode precursor is a composite electrode
20 precursor formed from a mixture comprising an active material, binder, and solvent then the preferred active material is a carbonaceous material such as carbon, the preferred binder is PVdF and the preferred solvents are N-methyl pyrrolidinone (NMP), dimethyl formamide (DMF),
25 and dimethyl acetamide (DMA).

Typically, the particles are applied to the surface of the electrode precursor such that they are in electrical contact with the intercalation material.

30

The process for preparing a conventional carbon composite anode typically takes the following general form. Carbon(s), binder and solvent are mixed together to achieve a uniform mix. The mixture is pattern coated onto
35 thin copper foil, with controlled evaporation of the solvent. The electrode is then dried (this may optionally

take the form of vacuum drying). The electrode is then calendered to achieve the required electrode porosity and then slit to the required electrode width. It is then vacuum dried, often at raised temperature and then
5 stored, usually as electrode reels (often vacuum packed), until required for cell construction.

The particles may be applied to the surface of a conventional carbon composite anode (electrode precursor)
10 at any suitable point during the preparation of the electrode precursor. Where the particles are lithium particles then application is preferably at any point after removal of the coating solvent. However, it is preferable to apply the particles after the electrode
15 precursor has been dried for the second time especially where the particles are lithium particles or stabilised lithium particles. This has various practical advantages. For example, calendering and slitting machines are not usually situated in areas with dry air,
20 and wet air will start to hydrolyse lithium particles. In addition, slitting generates waste material at the edges and ends of the coated material. If the whole reel is coated with particles, then those on the off-cuts are wasted. Further, calendering the electrode precursor
25 uses quite high pressures, whereas particles, and in particular lithium particles, only need light pressure to stick them to the electrode precursor. Although the particles will be integrated more uniformly into the electrode by calendering, the higher pressure may crack
30 open the stabilising layer of the stabilised lithium particles. This may lead to the lithium particles reacting as the freshly exposed lithium surfaces are very reactive. In addition, the particles may tend to stick to the metallic rollers of the calendaring machine, making
35 it difficult to control the quantity added, and requiring the rollers to be cleaned regularly.

The particles may be applied by any suitable method. Suitable 'wet' methods include spray coating of particles dispersed in liquid, metered pumping of a particle
5 dispersion onto the electrode surface, metered pumping of a particle dispersion using dissolved polymer to aid dispersion and particle adhesion, transfer roller coating using a particle dispersion or suspension, screen
10 printing of the particles dispersed in an 'ink', wet casting using "knife over plate" or "knife over roll", rotogravure or anilox roll coating, extrusion or slot-die coating (low pressure extrusion of a mixture of liquid and particles). Preferably the particles are applied
15 suspended in a liquid or are applied as a slurry or suspension. Suitable 'dry' methods include electrostatic transfer, cascade rolling of dry particles, or sprinkling the particles onto the electrode precursor surface using a sieve or 'pepper-pot' apparatus. Electrostatic transfer is a preferred 'dry' method.

20

After application the particles are typically fixed using light rolling. A protective sheet is usually required between the rollers and the electrode to prevent the particles from sticking to the rollers.

25

The particles may be applied to the surface of the electrode precursor so as to form a continuous or non-continuous coating. For example, the particles may be applied so as to cover only portions of the electrode
30 precursor.

A cell typically comprises at least an anode, cathode and an electrolyte. If the electrolyte is a liquid then, to ensure separation, a separator is
35 generally provided between the anode and the cathode in the cell. The separator may be a porous inert sheet for

example of glass fibre, polypropylene, or polyethylene. More preferably the separator is a polymeric sheet that forms a gel-like layer when impregnated by a non-aqueous solvent that acts as a plasticiser; desirably the sheet
5 is microporous. A suitable polymeric sheet comprises a polymer such as polyvinylidene fluoride (PVdF), or a copolymer of vinylidene fluoride with hexafluoropropylene (PVdF/HFP).

10 The particles may also be applied to the separator in the same way as to the electrode precursor where a separator is used in a cell. Accordingly, the present invention provides a process for producing a separator
15 for use in a cell comprising an intercalation material which process comprises forming a separator precursor and applying particles to the surface of the separator precursor wherein the particles comprise a material which intercalates into the intercalation material. The
20 present invention also provides separator for use in a cell comprising an intercalation material which separator comprises a separator precursor and a surface coating of particles comprising a material which intercalates into the intercalation material of the cell.

25 The separator precursor may be made of any suitable separator material including those described above.

The present invention also provides a cell comprising an electrode and/or separator of the present
30 invention and a battery incorporating one or more cells of the present invention.

One advantage of the present invention is that the particles applied to the electrode precursor or separator
35 precursor can compensate for the irreversible capacity of the cell, for example where the particles comprise

lithium particles. However, particles can also be applied to boost the overall capacity of the cell.

Specific constructions of cells comprising
5 electrodes embodying the invention will now be described by way of example and with reference to the drawings filed herewith, in which:

Figure 1 illustrates the cell voltages during the charge
10 and discharge cycle for the cells of example 1.
Figure 2 illustrates the cell voltages during formation and first discharge for the cells of example 2.

The present invention is now described in greater
15 detail by way of example.

Example 1

A lithium ion anode containing 91.5 % carbon and 8.5
20 % PVdF was coated on 10 μ m copper foil, using standard mixing and coating processes. A 40 mm diameter disc was cut from the calendered electrode, and placed in a glass beaker. Approximately 20 mg of stabilised lithium powder was weighed out into the beaker, and p-xylene was added
25 to make a slurry. The lithium was dispersed over the electrode, and the xylene was allowed to evaporate. After drying overnight in a vacuum oven at room temperature, the electrode was rolled lightly between two pieces of release paper. The lithium powder was visible on the
30 electrode surface, and the electrode weight had increased by 3.19 mg. The weight of carbon in the electrode was 105 mg. The lithium : carbon molar ratio was therefore around 0.05 : 1.

35 Discs were cut from the electrode with a 12.46 mm diameter cutter, and assembled into half cells with

lithium metal counter and reference electrodes. The electrolyte was 1.2 M LiPF_6 in a mixture of ethylene carbonate : ethyl methyl carbonate (2:8). The cells were cycled between 1.5 V and 0.005 V vs. Li/Li^+ , using a
5 current of ± 0.2 mA. As a control, electrodes were also cut from the original coated material, and assembled into half cells. The cell voltages during the initial charge and discharge cycle for one test cell and one control cell are shown in Figure 1.

10

The initial voltage of the test cell was 0.317 V vs. Li/Li^+ , compared to 2.712 V for the control cell. Lithium could also be extracted from the electrode in the test cell, before the initial charge. The coulombic efficiency
15 during the first cycle was 96.9 % for the test cell, and 81.2 % for the control cell. The capacity at around 0.8 V vs. Li/Li^+ that is associated with forming the SEI layer was absent in the test cell.

20

Figure 1 shows the initial charge and discharge of the test cell and the control cell. The control cell had an initial voltage of 2.712 V at a capacity of 0. As the cell was charged the capacity increased and the voltage decreased. This is due to the insertion of lithium into
25 the anode. Once the voltage reached 5 mV the process was reversed and the cell was discharged. During this process lithium moved out of the anode. At a capacity of about 70 mA hr g^{-1} the voltage suddenly increased. This was due to no more lithium being available. The
30 difference in capacity between the uncharged cell and the discharged cell was due to the irreversible capacity of the cell. In contrast, for the test cell the initial voltage was about 0.3 V. This was due to some prelithiation of the anode by the lithium powder.
35 Lithium was removed from the anode by discharging the cell. A charging and discharging cycle was then

performed in the same way as for the control cell. The voltage dropped to 5 mV earlier due to that fact that the solid electrolyte interface layer was already formed at the beginning of the experiment. However the capacity of the cell when discharged was much closed to that of the cell before charging started. This indicates that all the lithium that intercalated in to the anode on charging was also released by the anode on discharging. Thus, no lithium from the cathode was used to form the SEI layer as this was provided by the lithium powder. The small difference in capacity is likely to be due to internal resistance of the cell. These results all suggest that the lithium powder reacted to form an SEI layer on the carbon particles.

15

Example 2

Commercial lithium ion anode and cathode electrode materials were obtained from AGM Batteries Ltd., Thurso, Scotland. The anode was graphite/PVdF on copper foil, and the cathode was lithium cobalt oxide/carbon/PVdF on aluminium foil. Both electrodes had been calendered. The coating was removed from one side of each electrode, using N-methyl pyrrolidinone and gentle scraping. Smaller electrodes were then cut from each sheet; 32 x 51 mm for the anode and 30 x 49 mm for the cathode.

For the test cells, a measured quantity of lithium powder was applied to the surface of the anode. A mixture of stabilised lithium powder and carbon was dispersed in p-xylene, and a number of drops were applied using a syringe. The xylene was then allowed to evaporate before cell construction. The anodes in the control cells were not treated in any way.

35

One anode and one cathode were wrapped in a porous separator, and assembled into a soft pack cell. The cell was designed for a stack of several electrodes, so a plastic spacer was used to fill up the void space. The electrolyte was 1.2 M LiPF_6 in a mixture of ethylene carbonate : ethyl methyl carbonate (2:8). The cells were allowed to soak for around two hours, and then charged at 12 mA up to a capacity of 40 mA hr. After being left for three days to age, the cells were discharged at 8 mA down to 2.75 V. Figure 2 shows the charge and discharge voltages for one test cell and one control cell, during the initial cycle.

The voltage of the test cell before formation (first charge) was 2.733 V, compared to 0.010 V for the control cell. The test cell reached a higher voltage during formation, implying a higher state of charge. Charging of each cell stopped at a capacity of 0.04 A hr. The cells were then discharged. The test cell started at a higher voltage and produced more capacity during this subsequent discharge. This is shown in Figure 2. The coulombic efficiencies for this first cycle were 95.8 % for the test cell, and 92.0 % for the control cell. These results all suggest that the lithium powder reacted to form an SEI layer on the carbon particles.

Claims

1. A process for producing an electrode which comprises forming an electrode precursor comprising a layer comprising an intercalation material, and then applying particles to the surface of the electrode precursor wherein the particles comprise a material which intercalates into the intercalation material of the electrode.
2. A process according to claim 1 wherein the particles are lithium particles.
3. A process according to claim 1 or 2 wherein the particles are stabilised lithium particles.
4. A process according to any one of the preceding claims wherein the particles are applied to the anode.
5. A process according to any one of the preceding claims wherein the particles are suspended in a liquid for application to the electrode precursor.
6. A process according to any one of claims 1 to 4 wherein the particles are formed into a slurry or suspension and dispersed over the electrode precursor.
7. A process according to any one of claims 1 to 4 wherein the particles are applied by electrostatic transfer.
8. A process according to any one of the preceding claims wherein the particles are fixed to the electrode surface by rolling.

9. A process according to any one of the preceding claims wherein the particles are lithium powder mixed with carbon particles.

5 10. A process according to any one of the preceding claims wherein the electrode precursor is a composite electrode precursor comprising an active material and a binder, and prepared using a solvent for the binder.

10 11. A process according to claim 10 wherein the active material is carbon.

12. A process according to claim 10 or 11 wherein the binder is polyvinylidene fluoride (PVdF).

15

13. A process according to any of claims 10 to 12 wherein the process for producing the electrode precursor comprises the steps of

20 i) mixing the active material, binder and solvent together to achieve a uniform mix

ii) coating the mixture onto a thin copper foil, with controlled evaporation of the solvent

iii) drying the electrode

iv) calendaring the electrode, and

25 v) vacuum drying the electrode,

before applying the lithium powder to the electrode precursor.

30 14. An electrode comprising an intercalation material and a surface coating of particles comprising a material which intercalates into the intercalation material.

15. An electrode according to claim 14 wherein the particles are lithium particles.

35

16. An electrode according to claim 15 wherein the particles are stabilised lithium particles.

17. A process for producing a separator for use in a
5 cell comprising an intercalation material which process comprises forming a separator precursor and applying particles to the surface of the separator precursor wherein the particles comprise a material which intercalates into the intercalation material.

10

18. A process according to claim 17 wherein the particles are lithium particles.

19. A process according to claim 17 or 18 wherein the
15 particles are stabilised lithium particles.

20. A process according to any one of claims 17 to 19 wherein the particles are suspended in a liquid for application to the separator precursor.

20

21. A process according to any one of claims 17 to 19 wherein the particles are formed into a slurry or suspension and dispersed over the separator precursor.

22. A process according to any one of claims 17 to 19
25 wherein the particles are applied by electrostatic transfer.

23. A separator for use in a cell comprising an
30 intercalation material which separator comprises a separator precursor and a surface coating of particles comprising a material which intercalates into the intercalation material of the cell.

24. A separator according to claim 23 wherein the
35 particles are lithium particles.

25. A separator according to claim 24 wherein the particles are stabilised lithium particles.

5 26. A cell comprising an electrode produced according to any one of claims 1 to 13.

27. A cell comprising an electrode according to any one of claims 14 to 16.

10

28. A cell comprising a separator produced according to any one of claims 17 to 22.

29. A cell comprising a separator according to any one
15 of claims 23 to 25.

30. A battery comprising one or more cells according to any one of claims 26 to 29.

20

15794 TpCm

C.J. Talbot-Ponsonby
Agent for the Applicant

Abstract

Process for producing an Electrode

- 5 A process for producing an electrode which comprises
forming an electrode precursor comprising a layer
comprising an intercalation material, and then applying
particles to the surface of the electrode precursor
wherein the particles comprise a material which
10 intercalates into the intercalation material of the
electrode.

15794 TpCm

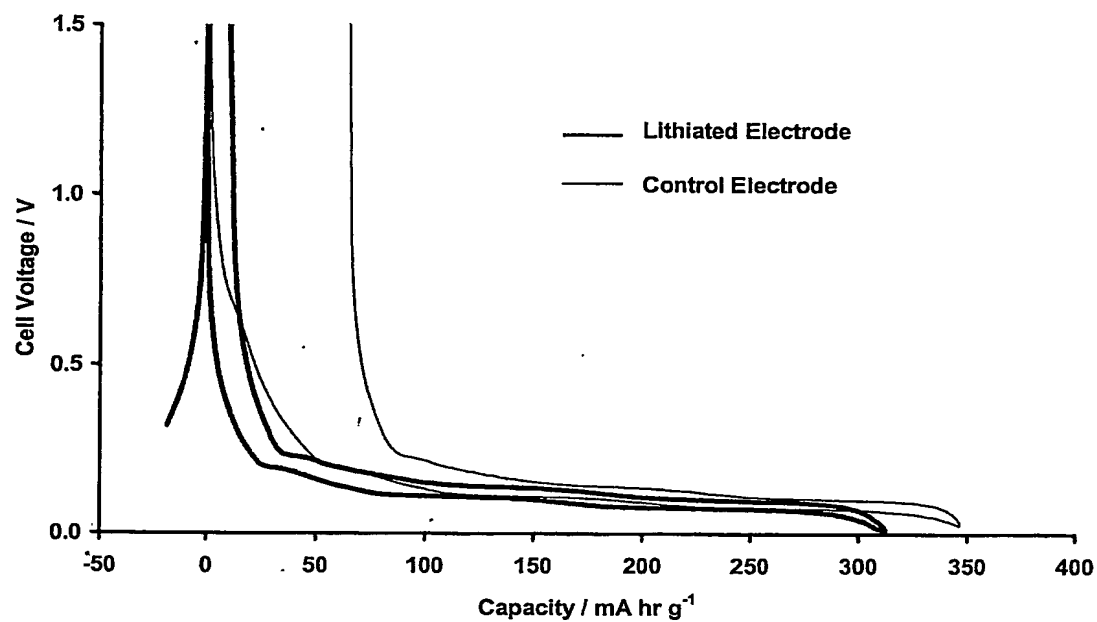


Figure 1.

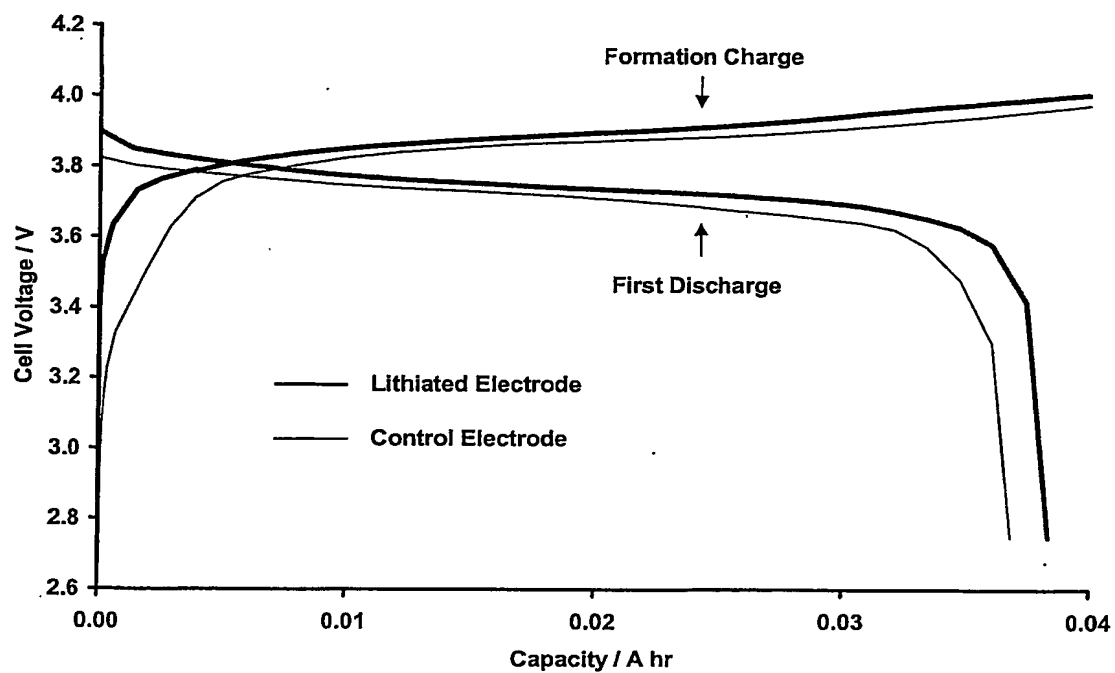


Figure 2.

Box No. VIII (iii) DECLARATION: ENTITLEMENT TO CLAIM PRIORITY

The declaration must conform to the standardized wording provided for in Section 213; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iii). If this Box is not used, this sheet should not be included in the request.

Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application specified below, where the applicant is not the applicant who filed the earlier application or where the applicant's name has changed since the filing of the earlier application (Rules 4.17(iii) and 51bis.1(a)(iii)):

In relation to this international application,

Accentus plc and FMC Corporation are entitled to claim priority of earlier application

No. GB 03 18942.0

by virtue of the following:

(ii) AEA Technology Battery Systems Limited was entitled as employer of the inventors:

LAIN Michael Jonathan
McDONALD Ian
BLACKMORE Paul David
DASS Vijay

(ii) FMC Corporation was entitled as employer of the inventor:

GAO Yuan

(iv) An assignment from AEA Technology Battery Systems Limited and FMC Corporation to Accentus plc and FMC Corporation dated 21 July 2004.

(ix) This declaration is made for the purposes of all designations.

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII

Box No. VIII (iv) DECLARATION: INVENTORSHIP (only for the purposes of the designation of the United States of America)

The declaration must conform to the following standardized wording provided for in Section 214; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iv). If this Box is not used, this sheet should not be included in the request.

**Declaration of inventorship (Rules 4.17(iv) and 51bis.1(a)(iv))
for the purposes of the designation of the United States of America:**

I hereby declare that I believe I am the original, first and sole (if only one inventor is listed below) or joint (if more than one inventor is listed below) inventor of the subject matter which is claimed and for which a patent is sought.

This declaration is directed to the international application of which it forms a part (if filing declaration with application).

This declaration is directed to international application No. PCT/..... (if furnishing declaration pursuant to Rule 26ter).

I hereby declare that my residence, mailing address, and citizenship are as stated next to my name.

I hereby state that I have reviewed and understand the contents of the above-identified international application, including the claims of said application. I have identified in the request of said application, in compliance with PCT Rule 4.10, any claim to foreign priority and I have identified below, under the heading "Prior Applications," by application number, country or Member of the World Trade Organization, day, month and year of filing, any application for a patent or inventor's certificate filed in a country other than the United States of America, including any PCT international application designating at least one country other than the United States of America, having a filing date before that of the application on which foreign priority is claimed.

Prior Applications:

I hereby acknowledge the duty to disclose information that is known by me to be material to patentability as defined by 37 C.F.R. § 1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the PCT international filing date of the continuation-in-part application.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

LAIN Michael Jonathan

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(city and either US state, if applicable, or country)

Mailing Address: c/o Accentus plc, Patents Dept

329 Harwell, Didcot, Oxfordshire, OX11 0QJ, United Kingdom

British

Citizenship:

Inventor's Signature: *Michael Jonathan Lain*
(if not contained in the request, or if declaration is corrected or added under Rule 26ter after the filing of the international application. The signature must be that of the inventor, not that of the agent)

Date: 5.18.04
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26ter after the filing of the international application)

MCDONALD Ian

Name:

Residence: Wantage, Oxfordshire, United Kingdom
(city and either US state, if applicable, or country)

Mailing Address: C/o Accentus plc, Patents Dept

329 Harwell, Didcot, Oxfordshire, OX11 0QJ, United Kingdom

British

Citizenship:

Inventor's Signature: *I. McDonald*
(if not contained in the request, or if declaration is corrected or added under Rule 26ter after the filing of the international application. The signature must be that of the inventor, not that of the agent)

Date: 5th Aug 2004
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26ter after the filing of the international application)

☒ This declaration is continued on the following sheet, "Continuation of Box No. VIII (iv)".

Continuation of Box No. VIII (i) to (v) DECLARATION

If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two inventors are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII ..." (indicate the item number of the Box) and furnish the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is needed in respect of two or more declarations, a separate continuation box must be used for each such declaration. If this Box is not used, this sheet should not be included in the request.

Continuation of Box No. VIII(iv)**Name:** BLACKMORE Paul David**Residence:** Abingdon, Oxfordshire, United Kingdom**Mailing Address:** c/o Accentus plc, Patents Dept, 329 Harwell, Didcot
Oxfordshire OX11 0QJ, United Kingdom**Citizenship:** British**Inventor's Signature:** **Date:****Name:** DASS Vijay**Residence:** Oxford, Oxfordshire, United Kingdom**Mailing Address:** c/o Accentus plc, Patents Dept, 329 Harwell, Didcot
Oxfordshire OX11 0QJ, United Kingdom**Citizenship:** British**Inventor's Signature:** **Date:** 05.08.04.

Continuation of Box No. VIII (i) to (v) DECLARATION

If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two inventors are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII ..." (indicate the item number of the Box) and furnish the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is needed in respect of two or more declarations, a separate continuation box must be used for each such declaration. If this Box is not used, this sheet should not be included in the request.

Continuation of Box No. VIII(iv)

Name: BLACKMORE Paul David

Residence: Abingdon, Oxfordshire, United Kingdom

Mailing Address: c/o Accentus plc, Patents Dept, 329 Harwell, Didcot
Oxfordshire OX11 0QJ, United Kingdom

Citizenship: British

Inventor's Signature: *PD Blackmore* Date: *11/8/04*

Name: DASS Vijay

Residence: Oxford, Oxfordshire, United Kingdom

Mailing Address: c/o Accentus plc, Patents Dept, 329 Harwell, Didcot
Oxfordshire OX11 0QJ, United Kingdom

Citizenship: British

Inventor's Signature: Date:

Continuation of Box No. VIII (i) to (v) DECLARATION

If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two inventors are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII ..." (Indicate the item number of the Box) and furnish the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is needed in respect of two or more declarations, a separate continuation box must be used for each such declaration. If this Box is not used, this sheet should not be included in the request.

Continuation of Box No. VIII(iv)

Name: GAO Yuan

Residence: Monroe, United States of America

Mailing Address: c/o Accentus pl, Patents Dept, 329 Harwell, Didcot
Oxfordshire OX11 0QJ, United Kingdom

Citizenship: American

Inventor's Signature: Date:.....

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